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TECHNIQUES FOR IMPROVING WEAR LIFE OF A SOLID-FILM LUBRICANT IN VACUUM

L. E. WIESER
B. D. McCONNELL

TECHNICAL REPORT AFML-TR-66-163

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AIR FORCE MATERIALS LABORATORY
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TECHNIQUES FOR IMPROVING WEAR LIFE OF A SOLID-FILM LUBRICANT IN VACUUM

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FOREWORD

This report was prepared by the Fluid and Lubricant Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, with L. E. Wieser and B. D. McConnell as Project Engineers. The work was initiated under project number 7343, "Aerospace Lubricants," Task Number 734302, "Aerospace Dry and Solid Film Lubricant Materials." This report covers the period of in-house work from 1 January 1964 to 31 December 1965.

The manuscript was released by the authors in May 1966 for publication as an RTD technical report.

This technical report has been reviewed and is approved.

R. L. Adamczak

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ABSTRACT

The objective of this program was to investigate methods for improving wear-life performance of a solid-film lubricant at elevated temperatures in a vacuum environment. The film formulation selected for this study was a MoS_2-PbS mixture bonded with a ceramic-metal oxide binder. The binder is composed of SiO_2, $NaCO_3$, Al_2O_3, Co_2O_3, CdO, CaO, $LiNO_3$, $K_2B_4O_7$:$5H_2O$ and $Ca(NO_3)_2$. Wear-life tests were conducted on a vacuum test rig to determine the degree of improvement. Test conditions were: pressures, approximately 10^{-6} torr; speed, 600 revolutions per minute; load, 25 and 52 pounds; temperature, 200° to 1000°F. Test specimens were a disk and two rub blocks. Only the disk was coated and was rotated between the two rub blocks placed 180° apart. Specimen materials were 4130, 4340, Rex AAA, M-10, 440C and René 41 alloys.

Three techniques were investigated to determine effectiveness for improving wear-life performance by producing a denser film. These included two methods of mechanical compressing: polishing and rolling the film. The third was a process of double deposition. Results were compared to conventionally sprayed films. The results indicate that polishing and rolling provide a very limited improvement but that the double-deposition method offers considerable wear-life improvement.

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INTRODUCTION

The objective of this program was to investigate methods for improving the wear-life performance of ceramic-bonded solid-film lubricants. This type of solid film is becoming more and more the preferable and sometimes necessary, method for lubricating moving parts in high temperature or otherwise hostile environments. Many of these films, however, do not possess adequate wear life for consideration in long-term operational systems, and continuous effort is being spent to develop new lubricating materials and better ceramic bonding compositions. Many of the compositions have properties which make them desirable for use in various applications but are passed over because of their short wear life.

This program was undertaken in an attempt to alleviate this problem by investigating methods of improving the wear-life performance of existing ceramic-bonded compositions without destroying other desirable properties such as adherence and good friction characteristics, and to investigate and establish the feasibility of techniques which may extend the wear-life performance of these types of films. Also, it was expected that additional knowledge of the behavior of ceramic-bonded films would be gained from these studies. In this report are presented the findings of these studies, and a discussion of the results and potential of the techniques investigated, which should provide a basis for further useful development of the most promising method.

SECTION II

SUMMARY

Three methods for providing a denser solid-film lubricant with some improvement in wear-life performance were tested and the results were compared to our normally sprayed films. This film, a MoS_2-PbS mixture bonded with a ceramic, metallic oxide frit, exhibits an average wear life of approximately 7,000 revolutions when normally sprayed, cured, and tested in vacuum (10^{-6} torr) at speeds of 600 rpm, loads of 25 pounds, and a temperature of 600°F.

The first improvement technique investigated was mechanical compressing by polishing the film with a quartz plate prior to testing. This technique indicated that a small increase in wear life of this film may be realized. The polished film exhibited an average of slightly over 23,000 revolutions for all tests or about three times that of the unimproved film.

The second technique studied was compressing the film by rolling with a special rolling device. This method differs from the polishing technique in that some of the film is not rubbed off and also orientation of the lubricant particles is minimal. Wear life with this technique is only somewhat higher (almost 38,000 revolutions) than with the polishing method. These two techniques do

not improve wear-life performance to the extent that the film could be considered useful in practical applications.

The most promising method studied was the third technique, that of double deposition. The use of electrophoresis to deposit a second lubricant-binder composition on the sprayed coating appears to densify the film by filling the voids and porous structure of the sprayed and cured film. Considerable improvement in wear life was noted when specimens prepared with this technique were tested under similar conditions. Test runs of over 1,000,000 revolutions were obtained although the average wear life exhibited by this technique was over 500,000 revolutions. Many applications of the sliding motion in a vacuum environment may be met by this film.

An attempt was made to determine the effect of heavier loads and higher temperatures on the performance of this film. Also, the effect of substrate materials was investigated. However, because of the limited number of tests conducted, these effects were not fully determined. Additional studies should be made with the double-deposition technique, with a sufficient number of tests conducted to establish repeatability, determine the above effects, and further verify the feasibility of improving wear-life performance with this technique.

SECTION III

APPROACH

Considerable thought and study was given to the various physical and chemical characteristics exhibited by ceramic-bonded solid-film lubricants. Various theories of friction wear, and lubrication were used to establish a theoretical background knowledge of the involved fundamental parameters (References 1, 2 and 3). The approaches taken, however, were limited to physical changes within the film structure.

Solid film formulations are applied to the substrate surface by various methods. Brushing, spraying, and dipping are among the most common methods used. The preferred method of applying ceramic-bonded films is spraying with a spray gun or airbrush. This method was chosen as the standard application technique for the purposes of this program. Experience has been developed with the spraying method and it was felt that more consistent coatings could be obtained than with other methods. Also the experience in evaluation of bonded films has been limited to the sprayed type.

Laboratory examination of normally sprayed ceramic-bonded films revealed them to be rather soft and powdery except at the metal-lubricant interface. Examination under a laboratory microscope showed these films to be very porous in nature with little bonding evident within the film. It was noticed in probing and examining the film that the porous structure could be compressed or compacted. The appearance of the film then was that of a more solid body which resisted further probing or moving. Also it was noticed that the surface of the film could be removed much easier than the material near the film-metal

interface. This same action had been noted in previous testing with other solid-film formulations in that the first few revolutions of the test specimen rubbed off the loosely adhering, powdery material until the remainder was compressed to a very thin film on the substrate surface. Thus, it seems that the ability of a solid film to lubricate under sliding conditions depends primarily on the production of a dense, well-bonded coating between the moving parts. The practice of keeping these coatings as thin as possible but with uniform quality has been generally accepted throughout the solid-film industry as the approach to follow.

These cursory examinations led to the idea that if techniques could be developed to provide denser, more uniform films, this perhaps would result in increased wear-life performance. A logical starting place seemed to be an attempt to produce the effect of run-in or the first few revolutions of a test which left the thin dense film. Therefore, mechanical compression of the film was selected as one approach. Two ways of accomplishing this became evident: compressing under sliding action or compressing under rolling action. A method of filling the porous structure with lubricant and binder was also felt to offer some benefit from the standpoint of a denser film. The method must be conducive to allowing particles to enter the voids and collect there rather than to building up a thicker film by particle deposit on the surface only. Electrophoresis, because of its tendency to deposit at the thinnest places, seemed most likely to meet this requirement (Reference 4). This technique, termed double deposition, was selected as the third area for investigation.

SECTION IV

PROCEDURES

SOLID-FILM FORMULATION

The effort devoted to this program was limited to the investigation of one solid-film-lubricant formulation. The film selected was a MoS_2-PbS mixture bonded with a ceramic, metallic oxide frit. The composition of the frit includes SiO_2, Na_2CO_3, Al_2O_3, Co_2O_3, CdO, CaO, $LiNO_3$, and $K_2B_4O_7 \cdot 5H_2O$. This material is prepared by mixing the above ingredients in the correct proportions, reacting them at 2200°F for about 10 minutes, then quenching by pouring the melted glass into cold water. This frit is then dried overnight in an air circulating oven and is ball-mill ground into powder. The binder and lubricant powders are sieved to pass through 325 mesh and are ready for use. The film formulation for these studies consisted of the following: oxide frit binder, 117 grams; MoS_2, 183 grams; PbS, 86 grams; $Ca(NO_3)_2$, 15 grams; and H_2O, 150 grams. The $Ca(NO_3)_2$ was added to bring the coefficient of thermal expansion closer to that of the substrate metals (Reference 5). The $Ca(NO_3)_2$ was dissolved in the 150 cc of H_2O and the solution, along with the other

powder, was placed in a pebble mill for 48 hours. After removal from the mill the resulting mixture was smooth and creamy. Additional water was added to thin the mixture to spraying consistency and stored until ready for use.

TEST SPECIMENS AND EQUIPMENT

The test specimens consisted of a disk or ring and two "rub" blocks, as shown in Figure 1. The test surfaces involved were the circumference of the ring or disk on which the coating is applied and the edges of the rub blocks. Specimen arrangement of the disk rotating between the two rub blocks situated 180° apart, is illustrated schematically in Figure 2. Specimen materials were 4130, 4340, Rex AAA, M-10, 440C, and René 41 alloys, which were varied as noted in the tables. The test surfaces were ground to a finish of 4 to 8 RMS. Hardness ranged from 42 to 52 Rockwell C.

All testing in this program was conducted with a vacuum friction and wear tester. This tester is shown in Figure 3, and has been described in detail in a previous report (Reference 7). Briefly, it consists of a rotating shaft through the base plate into the vacuum chamber. The opening is sealed with a rotating shaft seal which limits the vacuum to about 10^{-6} torr. The vacuum is maintained by a 6-inch oil diffusion pump. The shaft is connected directly to the drive motor which rests on a thrust bearing and is restrained from moving by a proof ring mounted with a linear variable differential transformer. Thus, the reaction torque is sensed by the LVDT and fed to a recorder which is calibrated to read friction force. The shaft has a cartridge heater located in the specimen area for heating the coated specimen and the rub shoe holders have three cartridge heaters to ensure even heating in the entire specimen area. Thermocouples in the end of the rub shoes register and control specimen temperature. The thermocouples are temperature sensitive approximately 1/8 inch from the rubbing surfaces. The temperature capability of the wear tester is ambient to 1200°F. Speed is variable from 50 to 800 rpm. The load is applied to the rub shoes through a series of lever arms and push rods through the base plate. These push rods are vacuum sealed with bellows. Loading is by a dead weight system with a 10 to 1 ratio: one pound on the load arm equals 10 pounds on each rub shoe. The load capability of the test rig is 10 to 800 pounds per shoe.

A rolling device, used initially to determine feasibility of the first method tested, is shown in Figure 4. It is a modified tubing cutter in which the cutting wheel was replaced with a steel roller. A second rolling device, designed and constructed during these investigations, is shown in Figure 5.

COATING AND CURING PROCEDURES

A standard pretreatment was established for the disk or ring specimens. This consisted of lightly sandblasting the surface to be coated, followed by cleaning thoroughly with Stoddard solvent and acetone, then placing the rings in an oven for oxidizing at 800°F for 10 minutes. The specimens were then ready for coating. Those not coated right away were stored in acetone until ready for spraying. Only the disk or ring specimens were subjected to the pretreatment and coating process. The rub blocks were used as received after cleaning with Stoddard solvent and acetone.

When the actual spraying procedure was begun, four to six cleaned specimens were placed on a shaft or mandrel. The shaft was driven by means of an electric motor with variable speed. The rotating speed of the specimen was maintained at approximately 300 rpm or at a surface velocity of 1.8 feet per second. An electric air heater was used to preheat the specimens on the shaft to a temperature of 150° to 180°F. Spraying was accomplished with an airbrush onto the continuous rotating specimens. Previously dispersed (in H_2O) the solid-film formulation was placed in the airbrush container. To achieve necessary spraying consistency, the mixture was continuously stirred with a magnetic spinbar. It was found that better spraying conditions existed if isopropyl alcohol diluted with 50 percent by volume of distilled water was added to the mixture. The air pressure supply to the airbrush was maintained at 55 pounds per square inch. The distance between the specimen and the nozzle of the airbrush was approximately 18 to 20 inches.

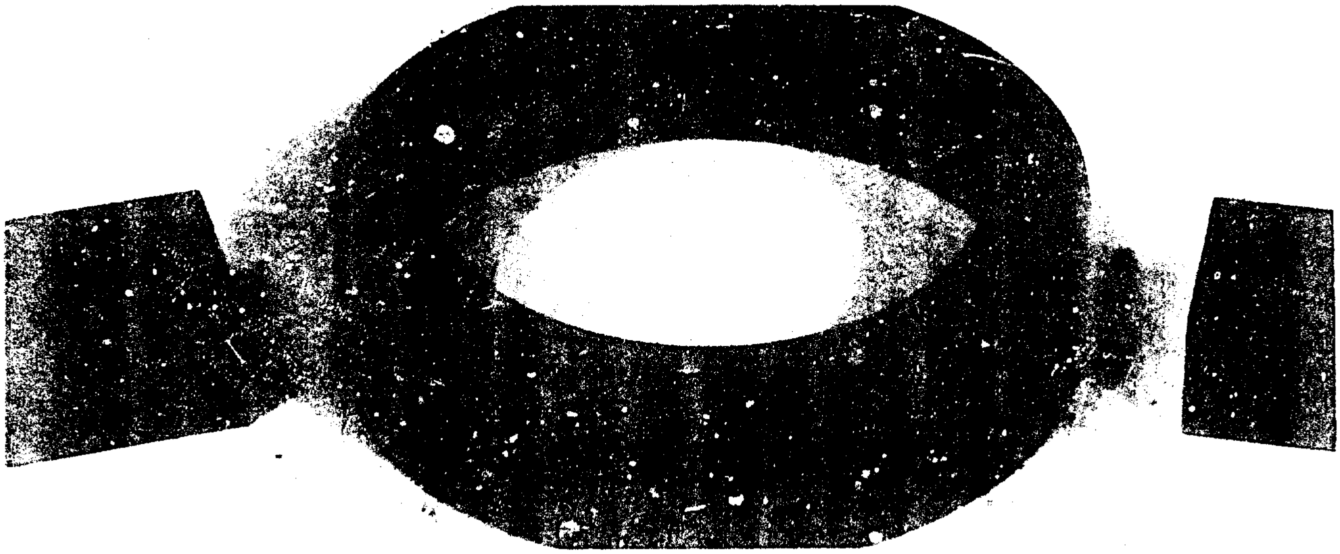

Figure 1. Test Specimen (Ring Type)

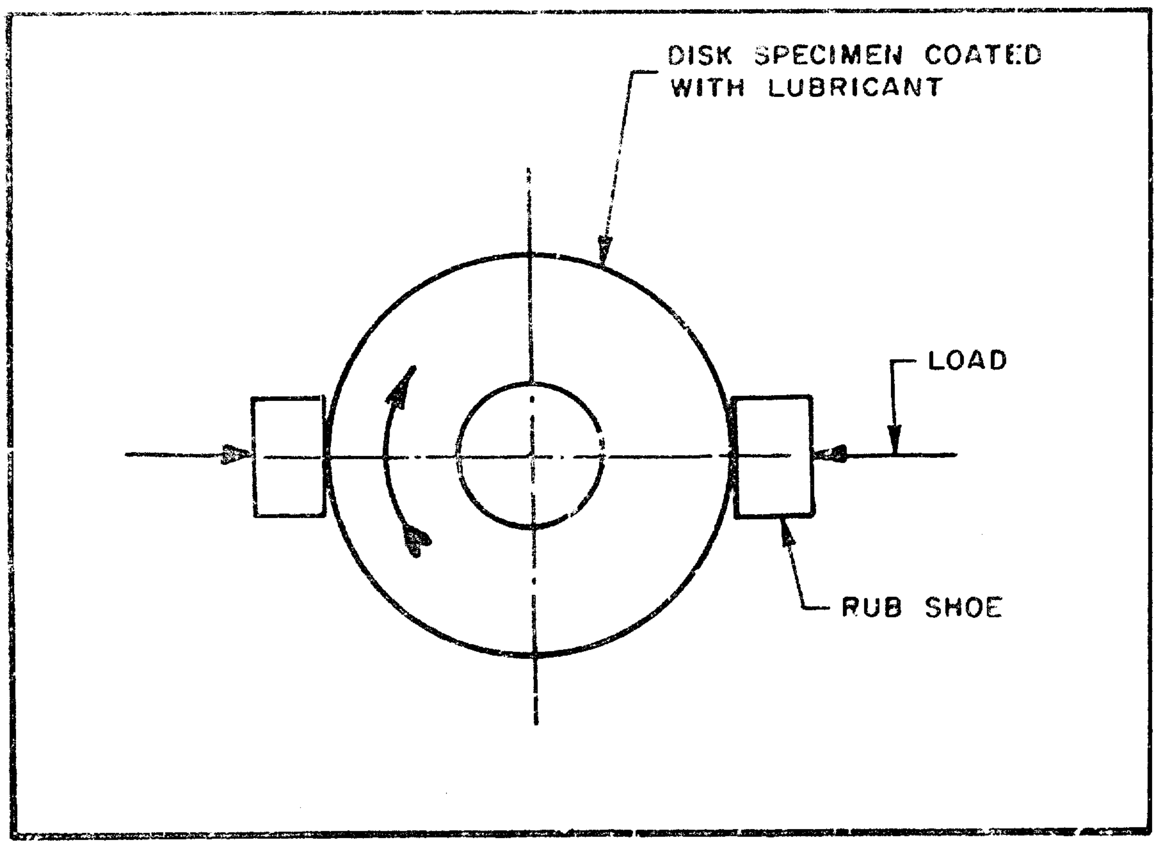

Figure 2. Arrangement of Test Specimen

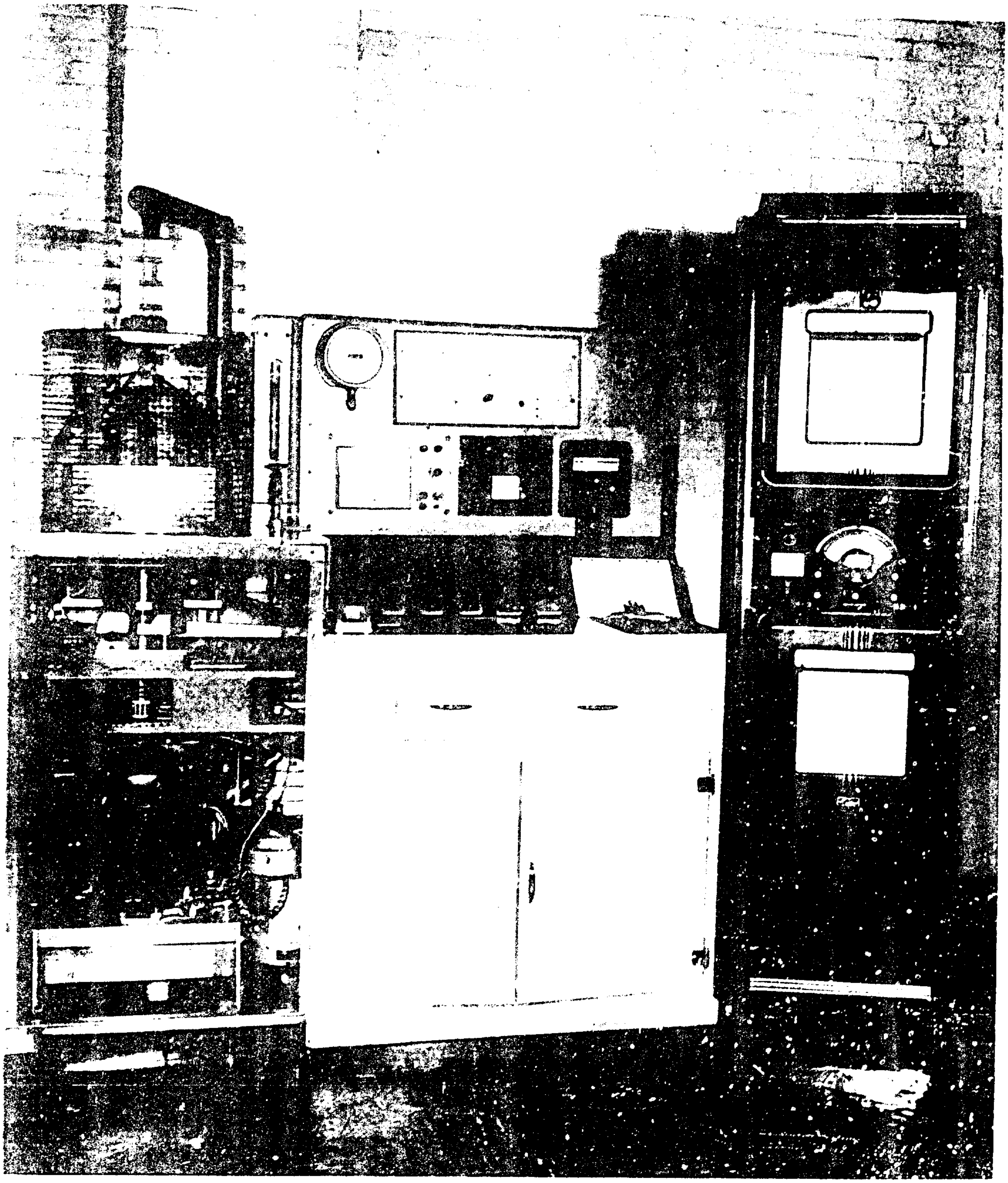

Figure 3. Vacuum Wear Tester

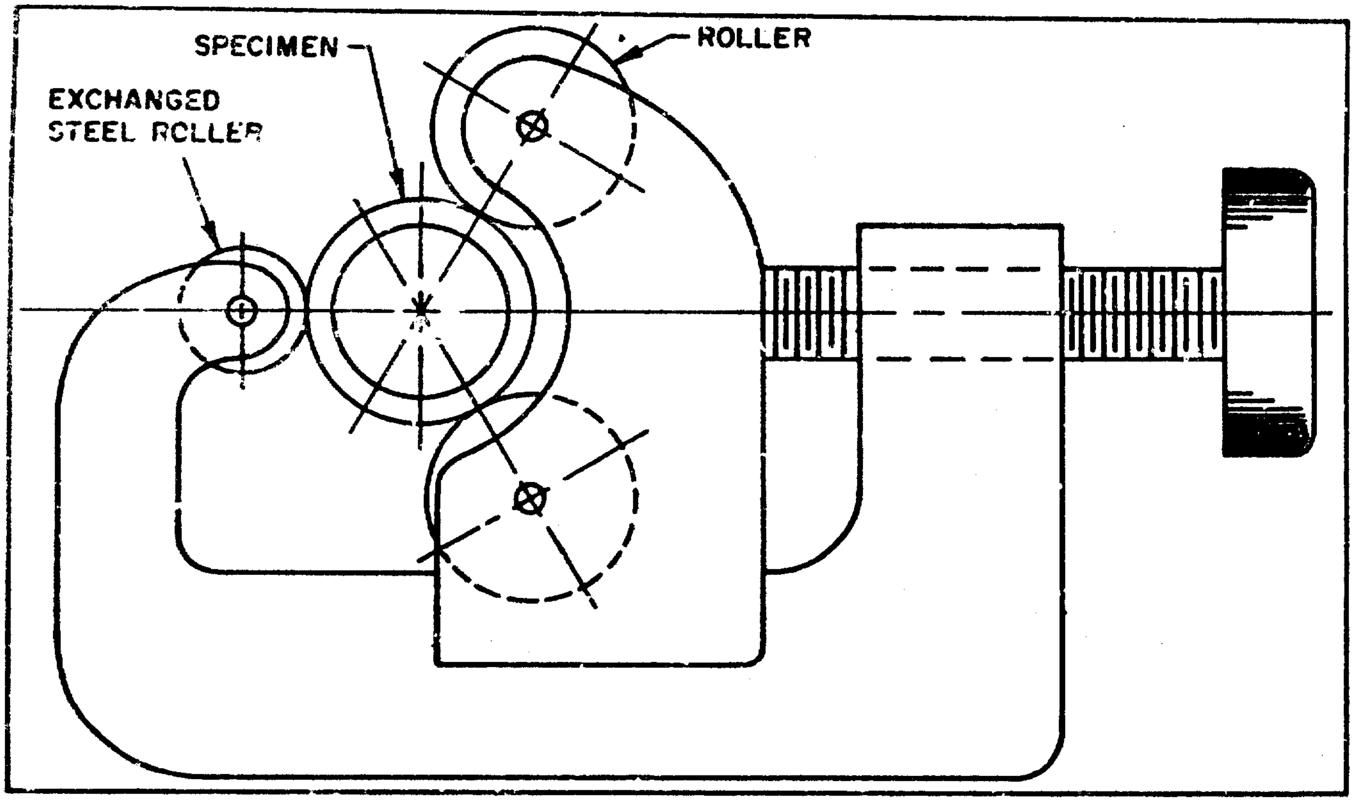

Figure 4. Rolling Device

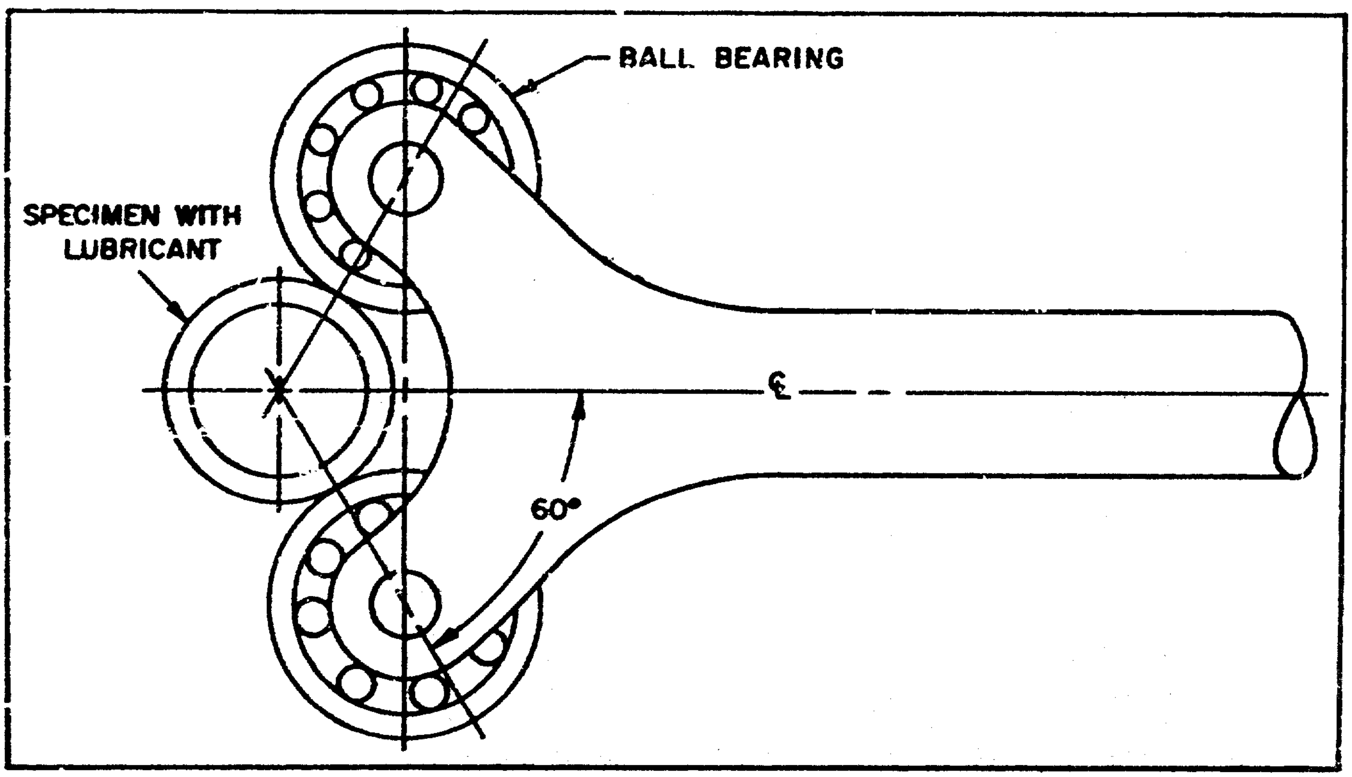

Figure 5. Improved Rolling Device

The spraying procedure was in four steps of some 20 to 30 seconds' duration each. Between each step was a time interval of some 3 to 5 minutes used for drying the applied solid-film lubricant. The drying process took place while the specimens were still rotating. The heat source was the electric air heater mentioned above. The color of the solid film appeared gray-blue. The general appearance was smooth, velvet-like, and soft. Under the laboratory microscope, however, voids and a non-uniform buildup of the particles were evident. This is shown in Figure 6, in which some of the particles are in sharp focus while others are not. Also, the voids appear as the dark spots in the picture.

The coated specimens were placed in a combustion oven (Reference 6) and heated slowly to 1450°F in a nitrogen atmosphere. The sintering process consisted of cycling the temperature between 1400° and 1450°F and holding the temperature for 5 minutes at 1450°F each time. This process was repeated four times. After the sintering process, the purging of nitrogen gas was continued until the temperature of the specimen was down to 250°F. The color of the film was still gray-blue. In the case where the second coating (MoS_2+B_2O_3) was applied to the first film layer, a second sintering process was performed in the same way as the first with the exception of temperature. In the second sintering temperatures of 1350° and 1400°F were used to fuse the B_2O_3 binder.

Upon cooling, the specimens were stored in a dessicator until needed for experimentation. Thickness of the applied solid films was measured with a Magnegage. Only the specimens responding to the magnetic system of the gage were used for measuring. These were the specimens made of type 4130 steel. The measurements were made after each sintering process of the film. The sprayed solid-film lubricant exhibited a thickness of .0019 to .0026 inches after curing. Measurements were taken also to determine the solid-film thickness on five specimens after the second solid-film lubricant was applied and sintered. These double-applied and twice-sintered solid films exhibited a total thickness ranging from .0045 to .0053 inch. The film thickness was fairly constant within this range when checked on randomly selected specimens and, therefore, each specimen was not measured.

No attempt was made to determine the film thickness after the test was run.

TEST CONDITIONS

Test conditions selected for these studies were the same as those used throughout many previous screening tests. This was considered an advantage in assessing the performance of this film. Test speed was maintained at 600 rpm or 215 feet per minute sliding velocity. The load was 25 pounds for most of the test although some deviation was made as noted in the tables. The load was applied slowly as the specimen rotated at test speed. The temperature was varied with some tests to determine the effect of temperature but most runs were made at 600°F. The best vacuum consistent with heat load was maintained and was around 10^{-6} torr most of the time. The coefficient of friction was monitored throughout the tests but only the friction at the start after run-in and at the finish of each test is reported. In some cases the starting or running friction was high and the test was allowed to run beyond the 0.15 arbitrarily selected friction coefficient cutoff point. These are noted in the tables.

MECHANICAL COMPRESSING TECHNIQUES

Polishing

Polishing as a mechanical compressing technique closely simulates the run-in period of bonded solid-film lubricants under sliding conditions (References 2 and 8). Most of the loose powdery, material is either compressed or worn off in the first few revolutions of a sliding test. Normally, friction then drops and the thin dense layer of film left on the substrate supports the load and provides the lubrication until the film is worn through. Also, orientation of the lubricant particles (References 2 and 9) by the sliding action may be another benefit derived from the run-in period. If this condition could be achieved by some mechanical action prior to testing, improved wear life should result.

A flat quartz plate approximately 3 inches in diameter and 1/2-inch thick was selected as the tool to perform the sliding compression, or polishing action. Quartz was selected because of its very smooth surface and the fact that it should not impart any harmful contamination in the form of wear debris during the polishing.

The coated specimen, after curing, was placed on an adapter and then placed in the chuck of a lathe. The specimen was rotated at 130 rpm. The flat surface of the quartz plate was pressed by hand against the coated portion of the specimen. The plate was held against the specimen for about 30 seconds and was repeated approximately five times. Material was worn off with the first application of the plate, as expected. Very little material was seen the second time and after that, none could be observed. The appearance of the film was that of a test before failure, a uniform shiny film and much denser than before. Figure 7 shows a micrograph of the polished film which appears smoother and does not have the voids noted in Figure 6. This picture was taken with half the exposure time of Figure 6 because of the greater reflectance of the polished surface.

Rolling

The rolling technique was tried as a method for compressing the film without removing as much material as noted with the polishing action. A suitable specimen holder was turned by means of an electric drill at 290 rpm. The specimen was held between the rollers as shown (Figure 4) and rolling pressure was applied by the threaded spindle. This device indicated that the cured film could be compacted by rolling but the technique had many shortcomings. Consequently, a second rolling device was designed and constructed. This uses two bearings as the rollers, affording smoother rolling (Figure 5). The improved rolling device was mounted in a small mechanical lathe in the position as shown. A suitable specimen adapter was placed in the chuck and supported by the live center in the tail stock. Vibration-free running made this version more suitable for performing the rolling action; the bearings were sealed to prevent any grease from coming into contact with the film; the rolling action appeared to produce a denser film; and, the shiny surface noted with the earlier polishing technique was also evident. But some blistering or peeling of the film was observed.

DOUBLE-DEPOSITION TECHNIQUE

Another method was conceived as a possible way to produce denser films without physically disturbing the sprayed and cured film on the specimen surface. It is referred to as a double-deposition technique because it employs the use of a second deposition of lubricant-binder material. Electrophoresis was selected as the method for applying this second deposition, primarily because of its unique ability for depositing particles at the thinnest portions of film first. It was felt that the voids present in the porous structure represented thin portions of the film and would perhaps be filled with both lubricating and binder particles. Thus, a denser, better bonded film would result from a second curing.

A small electrophoretic bath was used for application of the second deposition. This bath is described in detail in a previous report (Reference 10) and consists primarily of a power supply capable of 200 milliamperes of direct current at 1000 volts, a suitable specimen holder, and the bath holding the solution. A stainless steel beaker was used as the bath vessel which also permitted its use as either the anode or cathode; the specimen serves as the other electrode. The materials to be applied are sieved to 325 mesh and mixed in a nonconducting carrier. For these studies 20 grams of powder were mixed with 250 cc of 2-propanol. The work referenced in the above report had already established the voltage, time, and polarity parameters necessary to successfully apply several lubricant and binder materials. Molybdenum disulfide and boric oxide in the ratio of 3:1 by weight was chosen as the material for the second deposition, since polarity was the same for both materials and no difficulty was experienced in depositing them. The coated specimens were placed in the bath and held at 600 volts for 30 seconds. The specimens were then placed in the oven and cured in nitrogen gas in accordance with the sintering process described earlier.

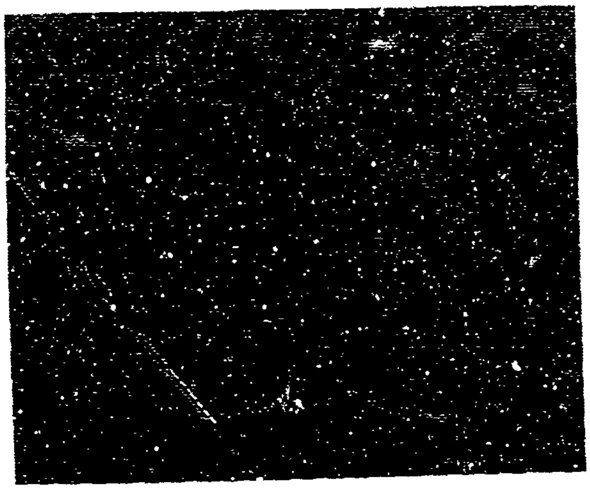

Figure 6. Micrograph of a Sprayed Film (1000X)

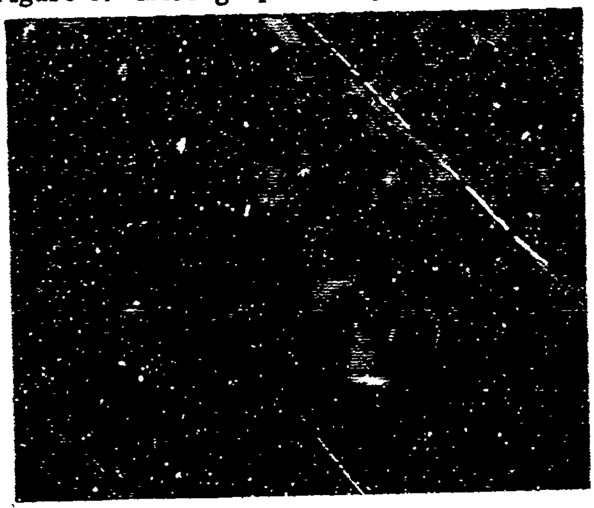

Figure 7. Micrograph of a Polished Film (1000X)

Figure 8. Micrograph of Double-Deposited Film (1000X)

The appearance of the film after curing was smooth, dark-gray, and somewhat thicker. Thickness measurements ranged from 0.0045 to 0.0053 inch for randomly chosen specimens. As shown in Figure 8 the surface texture of the film appeared more even and homogenous compared to the sprayed film in Figure 6. It appeared that many of the voids in the first coating were filled by the second deposition. The average particle size seems to be the same in both micrographs but many more particles are evident in the double-deposited film.

SECTION V
RESULTS

UNIMPROVED FILM

Additional specimens were prepared and tests conducted to supplement the base-line data available for comparison purposes. This was done to ensure comparable specimen pretreatment, spraying, parameters, and test conditions.

Table I shows the results of these tests. Disk and rub shoes of 4130 and René 41, respectively, were selected for these tests to eliminate any variables due to specimen materials. A friction coefficient of 0.15 was considered as the cutoff point for these tests. Some exceptions to this will be noted in later tables; however, comparisons will be limited to tests of like conditions. The average wear life for these seven tests is 7,173 revolutions, which is in the range normally exhibited by unimproved films of this type. The appearance of the test specimens from the first test in this table is shown in Figure 9. As noted in Table I, the temperature had risen to 820°F when the friction coefficient reached 0.15. Examination of the film under a laboratory microscope showed minute flaking with film wear, which could be seen under the microscope as fine parallel lines in the film.

POLISHED FILM

The wear-life data for the films treated with the polishing technique is shown in Table II. Tests 1 and 4, for some unknown reason, started with higher friction than was normal for this film and was allowed to run beyond the 0.15 cutoff point. The shorter lives of these tests may be attributed to the higher starting friction since it is believed that friction level influences wear-life performance of this film. This behavior is also noted in other data that follows. The problem noted in Test 2 was prevalent with a series of M-10 substrates at higher temperatures. This was traced to a fault in the bar stock from which these specimens were made. This test had not quite reached the 0.15 cutoff at the time the specimen cracked and the wear life would probably have been considerably longer if the test had been allowed to continue. The last test is shown because this was the only test conducted at a higher load with this technique. The effect on wear life cannot be substantiated since the test was not repeated. In fact, all of these tests were preliminary screening tests without attempts to repeat the conditions in many cases. However, since the factors noted generally would tend to shorten wear lives,

the polishing technique appears to offer limited improvement when compared to the values found in Table I. Wear life exhibited by this technique, based on these tests, is not of sufficient length to be considered useful for most applications.

ROLLED FILM

Table III contains the wear-life data for the tests conducted with the rolling technique. The average wear life of these tests is slightly higher than those with the polishing process and may offer a better means for compressing or densifying the film. The higher test temperatures (700° to 1000°F) did not appear to be detrimental in these tests. Also, the high load (100 pounds) does not drastically reduce the wear life based on Tests 2, 3, and 5. The improvement noted in Table III as compared to Table I may be due to the fact that the rolling process compresses the film without removing any significant amount of material as with the polishing technique. Again, short wear life is noted in Test 4 where the starting friction was higher for some unknown reason.

It is difficult to make a good valid comparison of the results in Tables I, II, and III because of the many variables and few tests. However, if it is assumed that specimen material has no appreciable effect on wear life and the effect of temperature can be neglected, then comparison of results would indicate some limited wear-life improvement can be obtained by mechanically compressing the film. This improvement, however, is not to the extent that these films can be considered useful in practical applications.

DOUBLE-DEPOSITED FILM

The technique of depositing a second coating on the first layer was the result of observing the normally sprayed and cured film to have voids which made the film porous and poorly bonded except at the metal-film interface. The results of tests conducted with specimens coated with a second deposit of lubricant-binder material are shown in Table IV. Considerable improvement in wear life over the other techniques is noted in these tests. These results show a great increase over the unimproved film and also over the polished or rolled film. This technique appears to have the ability to give consistently high average wear life compared to the other methods. Substrate material appears to have little effect on wear life although the 4130 steel alloy exhibited the best results. The tests running over one million revolutions are encouraging since this kind of wear life with the low friction level is in the range of being considered very useful in many applications. Figures 10 and 11 show the specimens after the completion of over 800,000 and 2,000,000 revolutions, respectively.

These results were felt to warrant further study of this technique and a second set of specimens were coated and tested under simiiar conditions except for higher load. Table V shows the results of these tests conducted with a 52-pound load. Again wear life was consistently high compared to the other techniques studied. The heavier load does not significantly affect wear life as tested under these conditions. The best results were obtained again with the 4130 alloy. Two of the tests, as noted, were stopped prior to failure because of noise, although the friction level was still very low. The noise was thought to be due to a loose locking nut, a defect which was known to have occurred in some prior tests. The tests were stopped to protect both the accumulated results and equipment. Also noted are the two tests with René 41 alloy which were not oxidized prior to coating and gave lower wear lives than in the other tests. This indicates the desirability of pre-oxidizing the substrate surface as part of the pretreatment procedure.

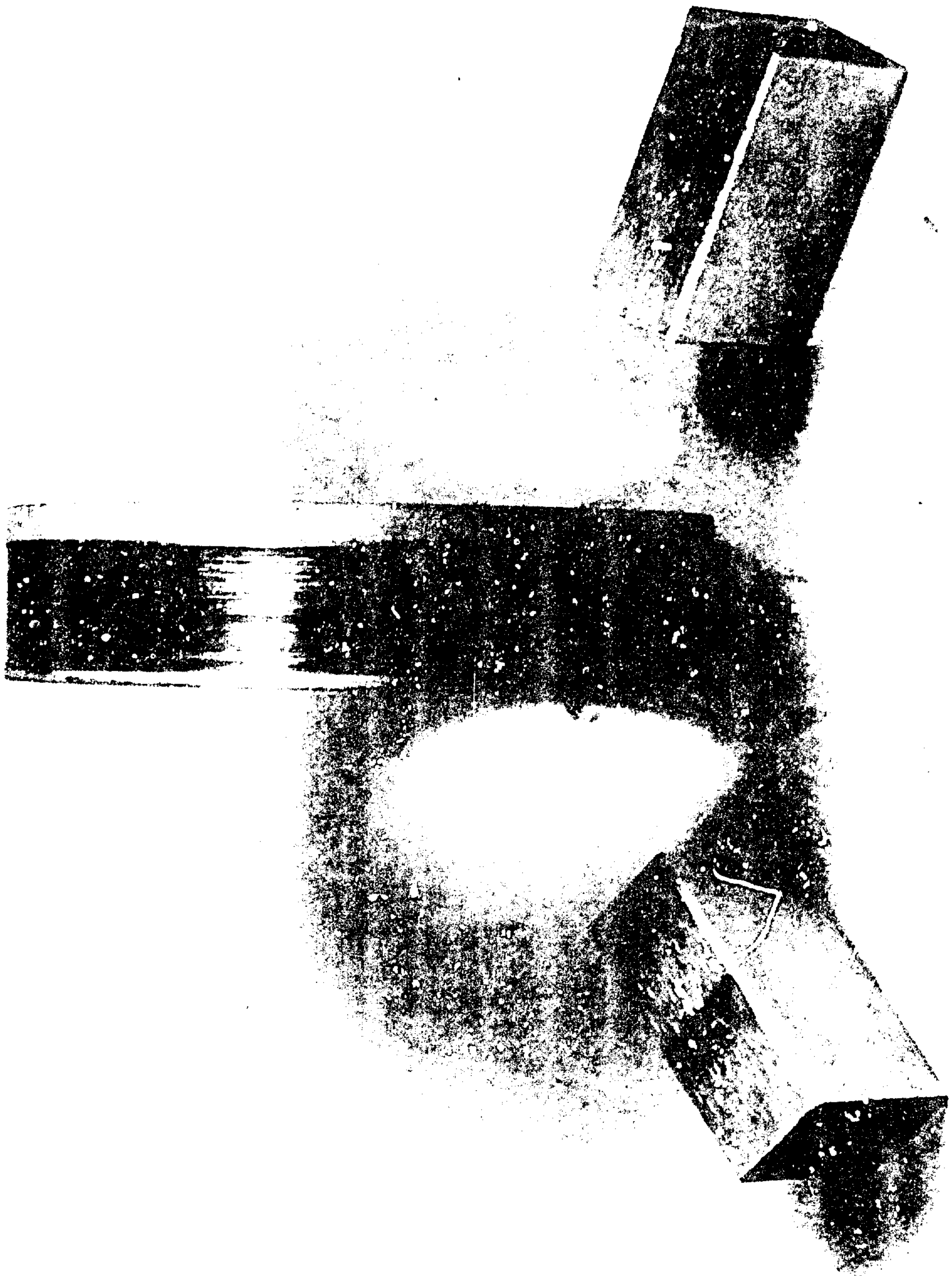

Figure 9. Specimen After 5,600 Revolutions

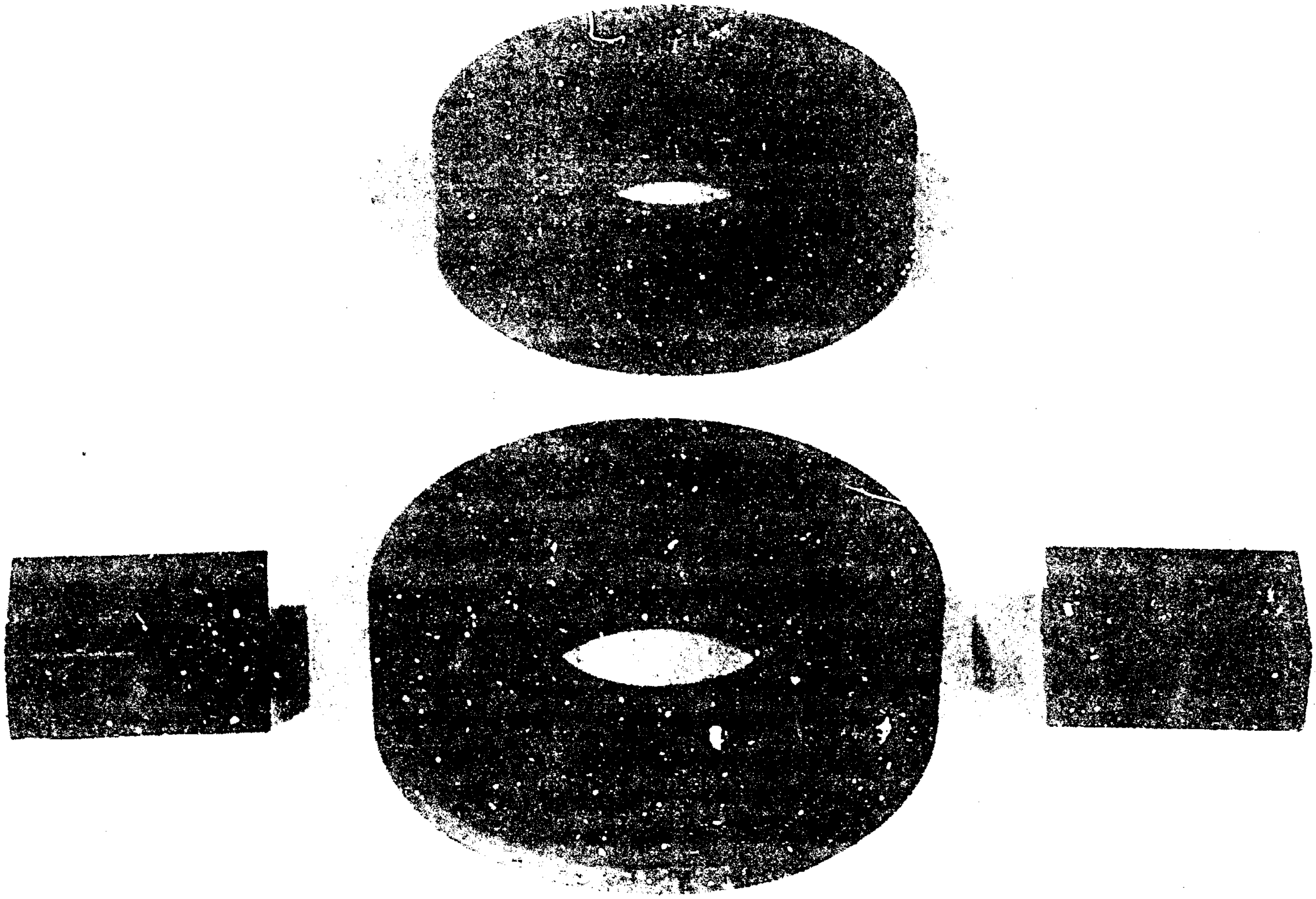

Figure 10. Double-Deposited Film with Over 800,000 Revolutions; Disk Type Specimen

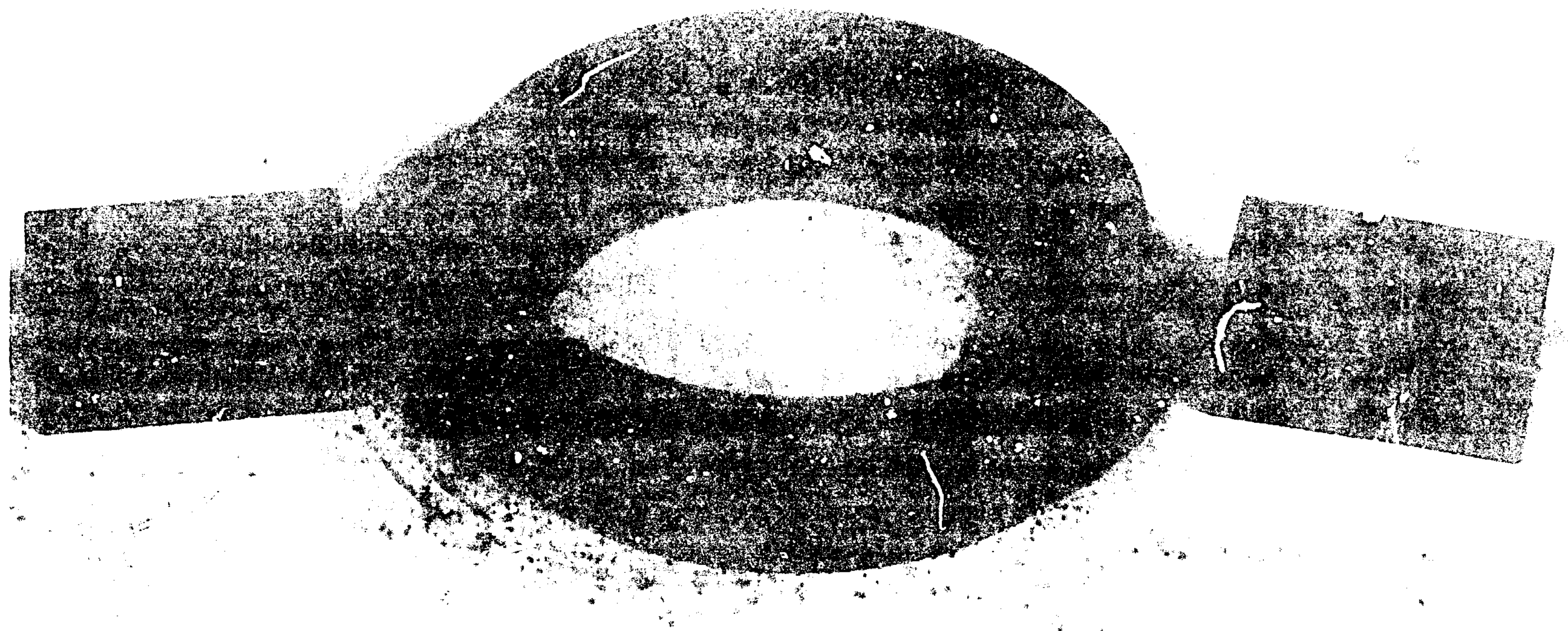

Figure 11. Double-Deposited Film with Over 2×10^6 Revolutions

TABLE I

WEAR-LIFE DATA OF UNIMPROVED FILMS

Pressure (Torr)	Friction Coefficient		Wear-Life Revolutions	Remarks
	Start	Finish		
8×10^{-6}	0.06	0.15	5,600	820°F @ cutoff
5.2×10^{-6}	0.04	0.15	4,383	
3.4×10^{-6}	0.04	0.15	13,648	
3.8×10^{-6}	0.05	0.15	14,792	
5.3×10^{-6}	0.04	0.15	5,212	
7.1×10^{-7}	0.06	0.15	4,577	
6.1×10^{-6}	0.04	0.15	2,013	

Test conditions: Speed, 600 rpm; load, 25 lbs/shoe; temperature, 600°F; rub shoe material, Rene 41; and, ring specimen material, 4130 steel.

TABLE II

WEAR-LIFE DATA OF THE POLISHED FILM

Specimen Disk	Material Shoe	Test Temp. (°F)	Pressure (Torr)	Friction Coefficient		Wear-Life Revolutions	Remarks
				Start	Finish		
Rex AAA	Rex AAA	750	8×10^{-7}	0.11	0.18	8,800	Friction high at start
M-10	M-10	1000	2×10^{-6}	0.08	0.13	40,500	Specimen cracked
M-10	M-10	750	2×10^{-6}	0.12	0.20	18,600	Friction high at start
4340	4340	650	5×10^{-7}	0.03	0.15	25,280	
4340	4340	600	3×10^{-7}	0.03	0.15	39,560	
440C	440C	580	6×10^{-7}	0.05	0.15	6,300	50-lb load

Test conditions: Speed, 600 rpm; load, 25 lbs/shoe.

TABLE III

WEAR-LIFE DATA OF THE ROLLED FILM

Specimen Disk	Material Shoe	Test Temp. (°F)	Pressure (Torr)	Friction Coefficient Start	Friction Coefficient Finish	Wear-Life Revolutions	Remarks
Rex AAA	Rex AAA	650	5×10^{-7}	0.08	0.15	41,000	At 15,600 Revolutions heater failed; finished at 450°F
Rex AAA	Rex AAA	710	3×10^{-7}	0.06	0.15	76,300	
M-10	M-10	700-980	5×10^{-7}	0.08	0.15	44,120	
M-10	M-10	1000	2×10^{-6}	0.15	0.30	8,600	Friction high at start
M-10	M-10	1000	4×10^{-6}	0.08	0.15	58,100	
René 41	René 41	500	5×10^{-6}	0.05	0.15	20,500	100-lb load
René 41	René 41	560	5×10^{-6}	0.06	0.15	25,000	100-lb load
4340	4340	600	5×10^{-7}	0.07	0.15	27,900	

Test conditions: Speed, 600 rpm; load, 25 lbs/shoe

TABLE IV

WEAR-LIFE DATA OF DOUBLE-DEPOSITED FILM WITH A 25-POUND LOAD

Specimen Material Disk	Pressure (Torr)	Friction Coefficient		Wear-Life Revolution	Remarks
		Start	Finish		
52100	6×10^{-6}	0.10	0.16	116,000	Substrate gold plated, not sand blasted
52100	4×10^{-6}	0.06	0.15	230,100	Substrate gold plated, not sand blasted
M-10	2.2×10^{-6}	0.06	0.15	42,300	
Rex AAA	2.6×10^{-6}	0.09	0.15	250,400	
Rex AAA	1.7×10^{-6}	0.09	0.15	131,650	
Rex AAA	1.1×10^{-6}	0.06	0.15	380,200	
4130	2×10^{-6}	0.06	0.15	415,800	
4130	1.5×10^{-6}	0.04	0.17	2,001,200	Figure 11 Test temperature 460°F
4130	1.5×10^{-6}	0.07	0.15	862,207	Figure 10 Test temperature 1000°F
4130	1.5×10^{-6}	0.10	0.25	537,704	Not [illegible]idized.
4130	1.5×10^{-6}	0.025	0.17	1,732,921	Test temperature 275°F

Test conditions: Speed, 600 rpm; load, 25 lbs/shoe; temperature, except as noted, 600°F; and rub shoe material, René 41.

TABLE V

WEAR-LIFE DATA OF DOUBLE-DEPOSITED FILM WITH A 52-POUND LOAD

Specimen Material Disk	Pressure (Torr)	Friction Coefficient		Wear-Life Revolution	Remarks
		Start	Finish		
René 41	1.5×10^{-6}	0.09	0.15	278,206	Not oxidized
René 41	1.4×10^{-6}	0.08	0.15	317,000	
René 41	1.8×10^{-6}	0.08	0.15	335,000	
René 41	2.2×10^{-6}	0.10	0.15	204,050	Not oxidized
4130	2.0×10^{-6}	0.08	0.15	552,043	
4130	2.1×10^{-6}	0.02	0.15	248,380	
4130	2.8×10^{-6}	0.075	0.065	600,000	Terminated because of noise
4130	2.0×10^{-6}	0.065	0.08	700,000	Terminated because of noise
M-10	2.0×10^{-6}	0.075	0.15	473,000	
M-10	2.0×10^{-6}	0.06	0.16	367,400	

<u>Test conditions</u>: Speed, 600 rpm; load, 52 lbs/shoe; temperature, 600°F; and rub shoe material, René 41.

SECTION VI

CONCLUSIONS

1. Densification of the film by the double-deposition technique offers the most potential for improving wear-life performance.

2. Based on the results of the limited tests with this technique, we may expect the the film to provide suitable operation for a moderately loaded journal or spherical type of bearing operating in a vacuum environment. However, further detailed testing should be done to establish repeatability and confidence limits.

3. Other normally sprayed ceramic-bonded films may be improved with the double-deposition technique since the voids and non-uniform films are believed to be the result of spraying and curing and not film composition. Also, choice of substrate materials should not significantly limit the use of this technique.

4. Mechanically compressing the film does not extend wear life to the degree offered by the double-deposition technique. Wear lives obtained from polishing or rolling the film are not considered useful for widespread applications.

SECTION VII

FUTURE WORK

1. The double-deposition technique for densifying films will be studied with other film formulations. This will establish the feasibility of the technique to work satisfactorily with different lubricant-binder materials as well as with other metal substrates.

2. Other substrate pretreatments and coating parameters will be studied to establish an optimum method. This will allow selection of a standard procedure to follow.

3. A technique for improving performance of high temperature, solid-film lubricants for oxidizing environments should be investigated and developed. This study should include bearing tests, with the films on spherical or journal type bearings, to correlate data as well as to establish the feasibility of improving performance in actual bearing assemblies.

SECTION VIII

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13. ABSTRACT

The objective of this program was to investigate methods for improving wear-life performance of a solid-film lubricant at elevated temperatures in a vacuum environment. The film formulation selected for this study was a MoS_2-PbS mixture bonded with a ceramic-metal oxide binder. The binder is composed of SiO_2, $NaCO_3$, Al_2O_3, Co_2O_3, CdO, CaO, $LiNO_3$, $K_2B_4O_7{:}5H_2O$ and $Ca(NO_3)_2$. Wear-life tests were conducted on a vacuum test rig to determine the degree of improvement. Test conditions were: pressures, approximately 10^{-6} torr; speed, 600 revolutions per minute; load, 25 and 52 pounds; temperature, 200° to 1000°F. Test specimens were a disk and two rub blocks. Only the disk was coated and was rotated between the two rub blocks placed 180° apart. Specimen Materials were 4130, 4340, Rex AAA, M-10, 440C and René 41 alloys. Three techniques were investigated to determine effectiveness for improving wear-life performance by producing a denser film. These included two methods of mechanical compressing: polishing and rolling the film. The third was a process of double deposition. Results were compared to conventionally sprayed films. The results indicate that polishing and rolling provide a very limited improvement but that the double-deposition method offers considerable wear-life improvement.

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Vacuum Environment						
Ceramic-Metal Oxide Binder						
Mechanical Compressing						
Polishing						
Rolling						
Double-Deposition						
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